

# New Advancements in Preparing Solid and Semi Solid Samples for POPs Analysis

Matthew MacLennan – Pacific Rim  
Laboratories

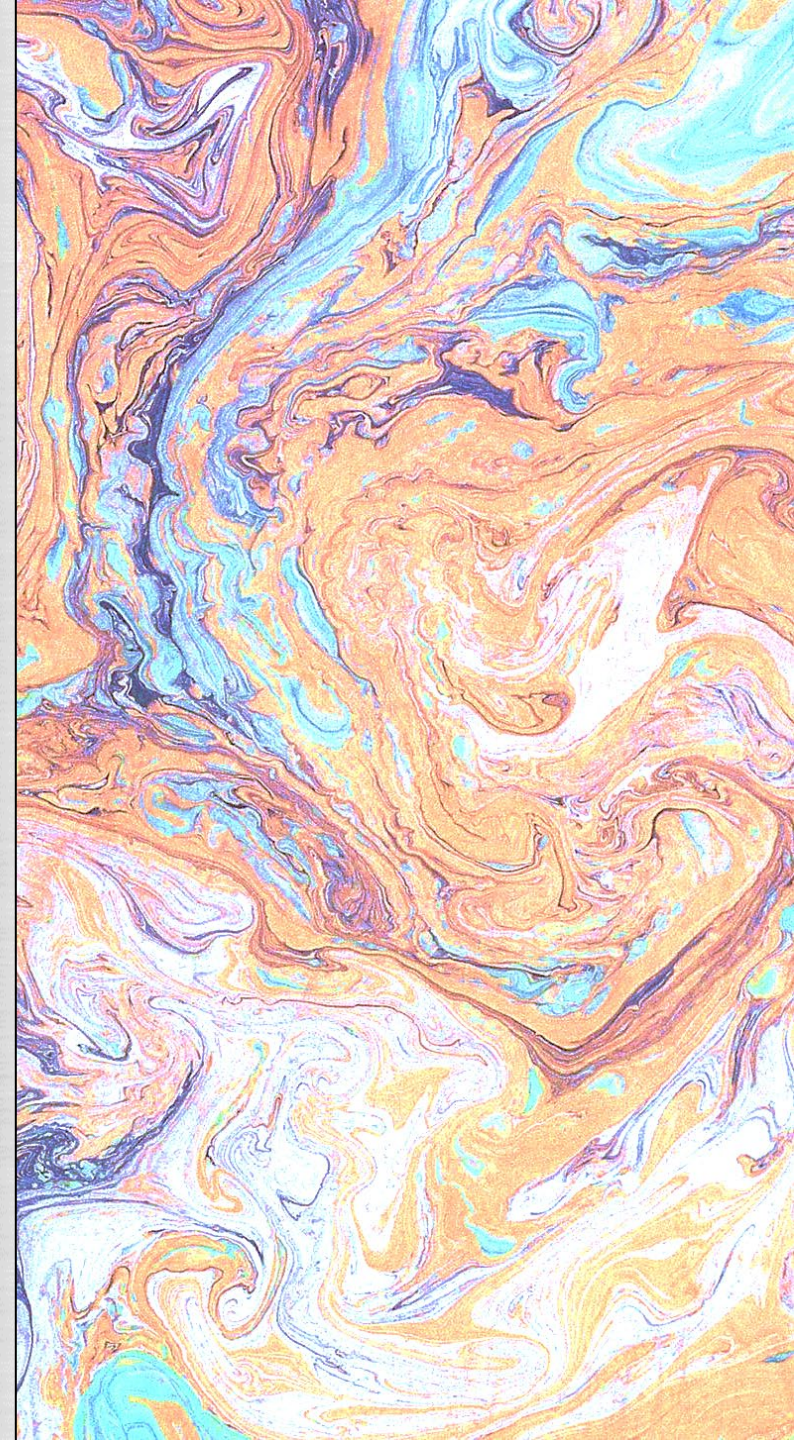
Chris Shevlin – Thermo Fisher Scientific



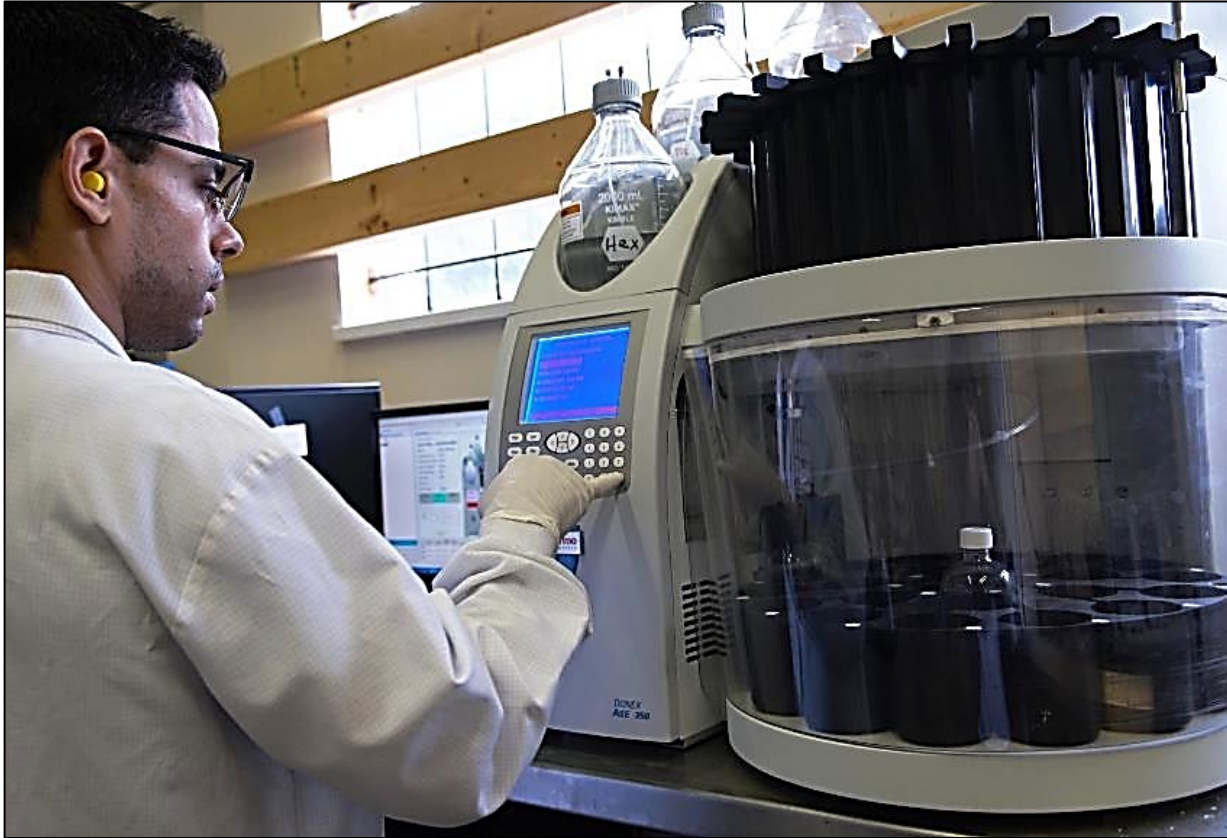
# EXTREVA ASE

Up-to-date Data

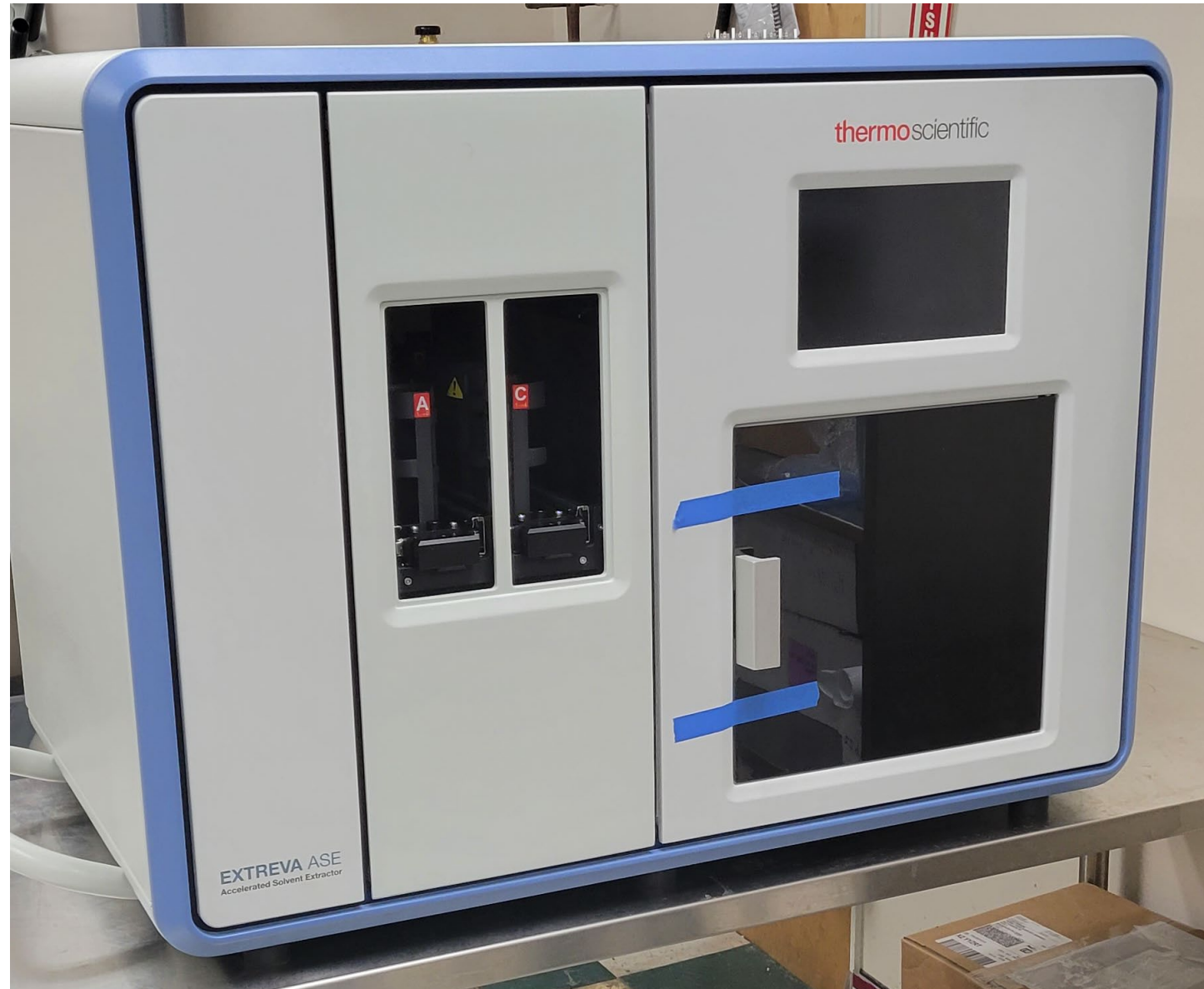
Pacific Rim Laboratories



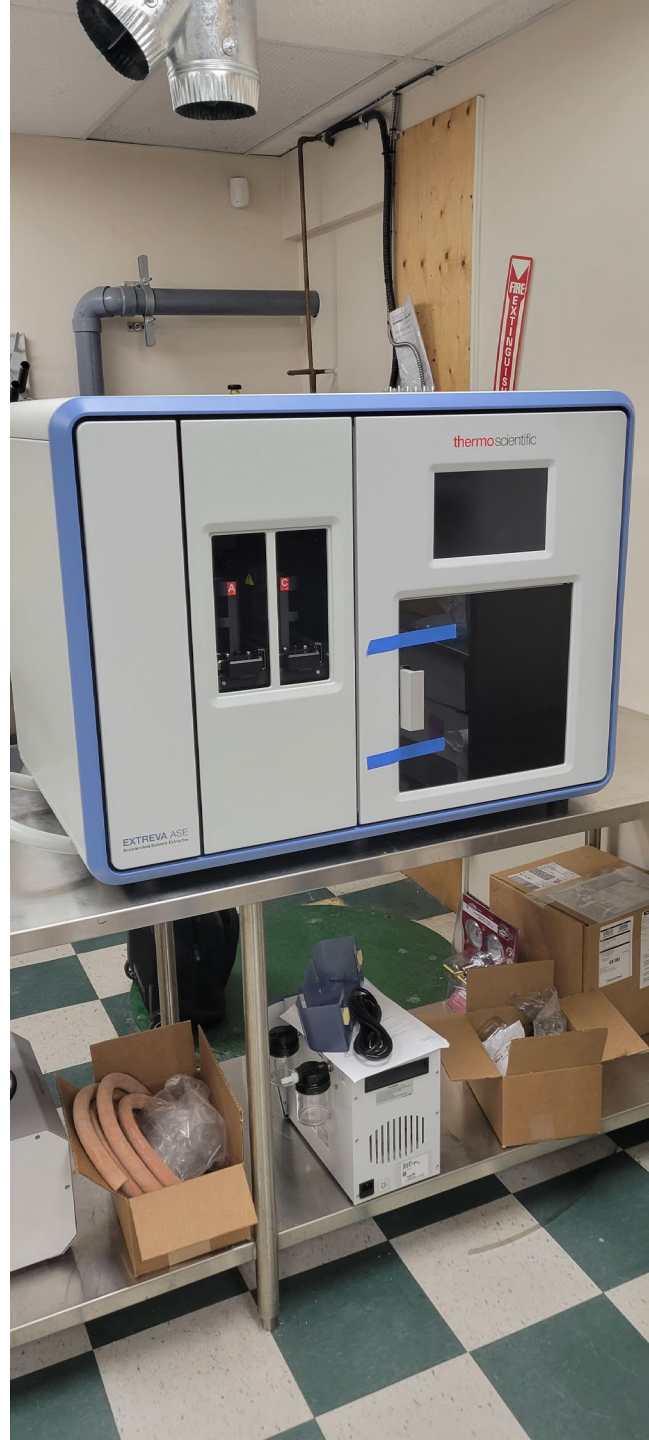
# ASE 350 + Rocket Evaporator



# Extreva ASE







# Table of Contents

1. Tests → DxF/PCB and PFAS
2. Tests on ASE350 versus Extrema
3. Method Transfer Results
4. Conclusions and Recommendations



# Extreva Specs

Test		Temp (°C)	Fill vol	Static phases	flow rate	control flow time	Extraction time	Comments
DF/PC	<b>EXTREVA</b>	100	50%		5.7 mL/min	20 mins	0:53:13	all four bottles have ~ 160 mL extracts
DF/PC	<b>EXTREVA</b>	100	50%		1.5 mL/min	20 mins	0:50:52	Low extracts volume ~40-50ml instead of 85 mL , especially 3210B with less than 10 mL extract collected
DF/PC	<b>ASE 350</b>	100	70%	2 x 5 mins			2:08:00	as usual, nothing special
DF/PC			50%	3 x 5 mins				
DF/PC	<b>EXTREVA</b>	100	50%		1.5 mL/min	20 mins	0:52:35	3218B from channel 1 also has low extract, re-extracted once more the next day .



# **Test Summary: Dioxin/Furan; PCB → *soil***

Two tests on Extrema

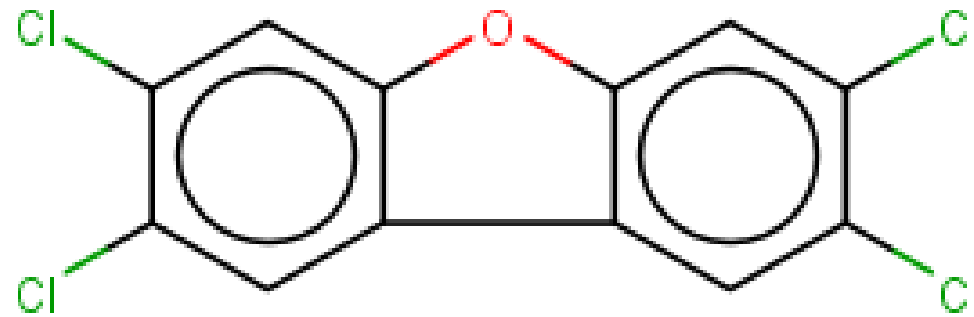
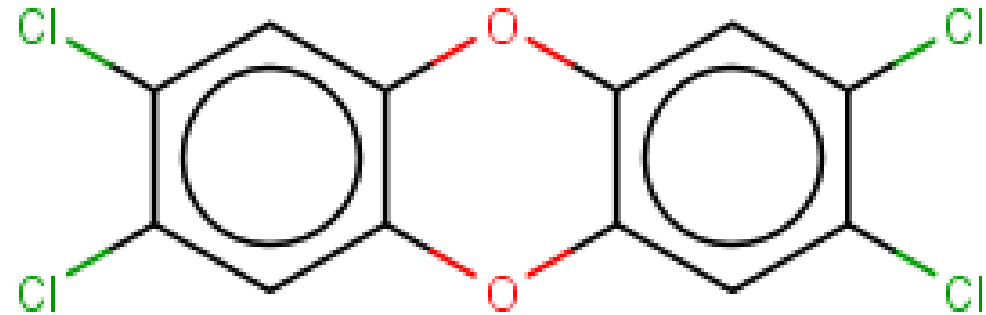
1. ASE350 method direct transfer
2. ASE350 method using half the solvent volume



# Test Summary: Dioxin/Furan; PCB → *soil*

Analysis of  
- 7 dioxins  
- 10 furans

2378-TCDD
12378-PeCDD
123478-HxCDD
123678-HxCDD
123789-HxCDD
1234678-HpCDD
OCDD
-
2378-TCDF
12378-PeCDF
23478-PeCDF
123478-HxCDF
123678-HxCDF
234678-HxCDF
123789-HxCDF
1234678-HpCDF
1234789-HpCDF
OCDF





# GC-HRMS Analysis DxF in soil

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# ASE 350 optimized method

INTERNAL STANDARDS	% REC	STDEV	RSD
2378-TCDD	50	5.57	11.2%
12378-PeCDD	68	5.32	7.8%
123478-HxCDD	72	4.55	6.3%
123678-HxCDD	105	10.02	9.6%
1234678-HpCDD	72	10.86	15.1%
OCDD	40	2.83	7.1%
2378-TCDF	44	5.44	12.4%
12378-PeCDF	51	0.82	1.6%
23478-PeCDF	60	6.45	10.8%
123478-HxCDF	68	8.29	12.2%
123678-HxCDF	95	6.90	7.2%
234678-HxCDF	94	9.00	9.5%
123789-HxCDF	86	4.03	4.7%
1234678-HpCDF	93	7.63	8.2%
1234789-HpCDF	74	8.46	11.5%



# GC-HRMS Analysis DxF in soil

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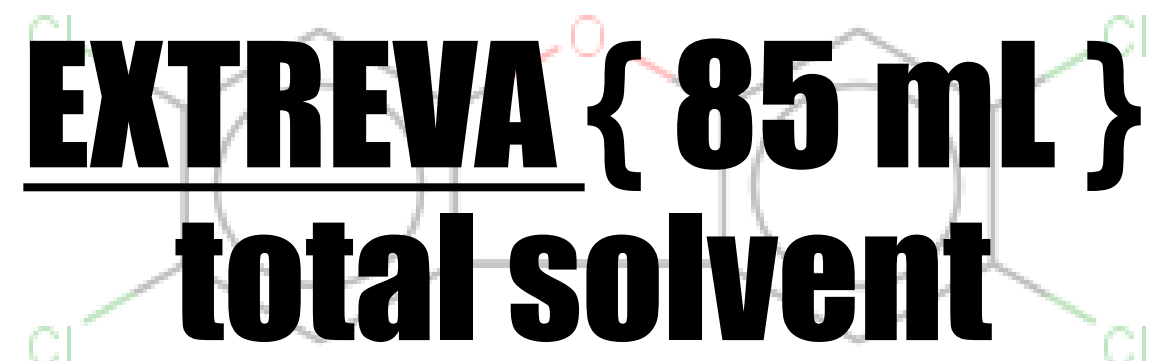
**EXTREVA { 170 mL }**  
**total solvent**

INTERNAL STANDARDS	%REC	STDEV	RSD
2378-TCDD	44	4.65	10.5%
12378-PeCDD	61	5.51	9.1%
123478-HxCDD	59	7.70	13.1%
123678-HxCDD	82	10.87	13.2%
1234678-HpCDD	57	5.97	10.4%
OCDD	35	4.65	13.5%
2378-TCDF	37	5.45	14.9%
12378-PeCDF	46	6.32	13.7%
23478-PeCDF	52	5.19	9.9%
123478-HxCDF	54	4.11	7.6%
123678-HxCDF	77	9.36	12.1%
234678-HxCDF	76	5.35	7.0%
123789-HxCDF	60	6.55	10.9%
1234678-HpCDF	72	7.27	10.1%
1234789-HpCDF	59	7.27	12.3%



# GC-HRMS Analysis DxF in soil

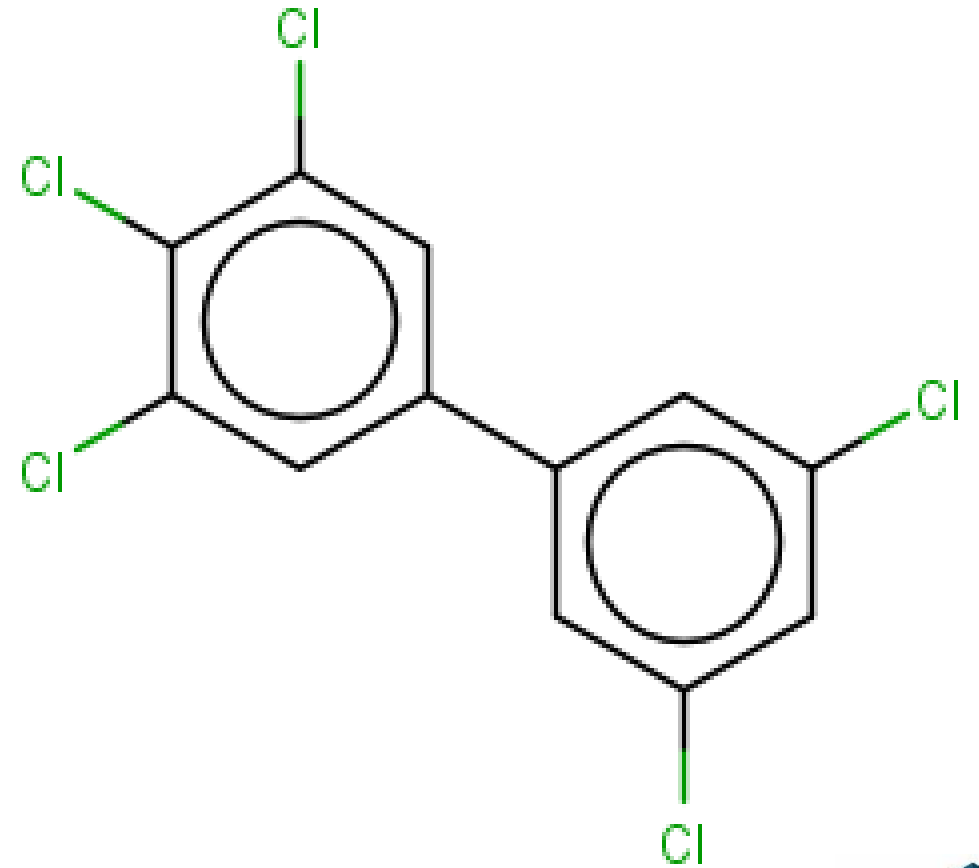
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## EXTREVA { 85 mL } total solvent

INTERNAL STANDARDS	%REC	STDEV	RSD
2378-TCDD	30	0.5	1.7%
12378-PeCDD	38	4.65	12.1%
123478-HxCDD	41	6.85	16.8%
123678-HxCDD	54	8.54	15.9%
1234678-HpCDD	37	2.94	8.0%
OCDD	23	4.69	20.4%
2378-TCDF	26	2.52	9.9%
12378-PeCDF	31	3.10	9.9%
23478-PeCDF	37	3.20	8.7%
123478-HxCDF	38	4.55	12.0%
123678-HxCDF	51	8.54	16.7%
234678-HxCDF	49	6.35	13.1%
123789-HxCDF	44	7.18	16.4%
1234678-HpCDF	44	5.74	13.2%
1234789-HpCDF	39	3.30	8.4%

# GC-HRMS Analysis of 209 PCBs in soil



# GC-HRMS Analysis of 209 PCBs in soil ASE 350 Method – Std Recoveries

C13 INTERNAL STANDARD	%REC	RSD
PCB 1	7	33%
PCB 3	8	45%
PCB 4	13	30%
PCB 15	17	36%
PCB 19	20	25%
PCB 37	34	21%
PCB 54	27	21%
PCB 81	59	10%
PCB 77	64	10%

PCB 104	32	14%
PCB 123	62	5%
PCB 118	60	6%
PCB 114	62	4%
PCB 105	66	3%
PCB 126	77	4%
PCB 155	40	11%
PCB 167	65	3%
PCB 156	68	7%

PCB 157	70	2%
PCB 169	66	5%
PCB 188	50	3%
PCB 189	87	7%
PCB 202	55	3%
PCB 205	74	1%
PCB 208	70	8%
PCB 206	61	3%
PCB 209	60	3%

# GC-HRMS Analysis of 209 PCBs in soil EXTREVA Method – Std Recoveries

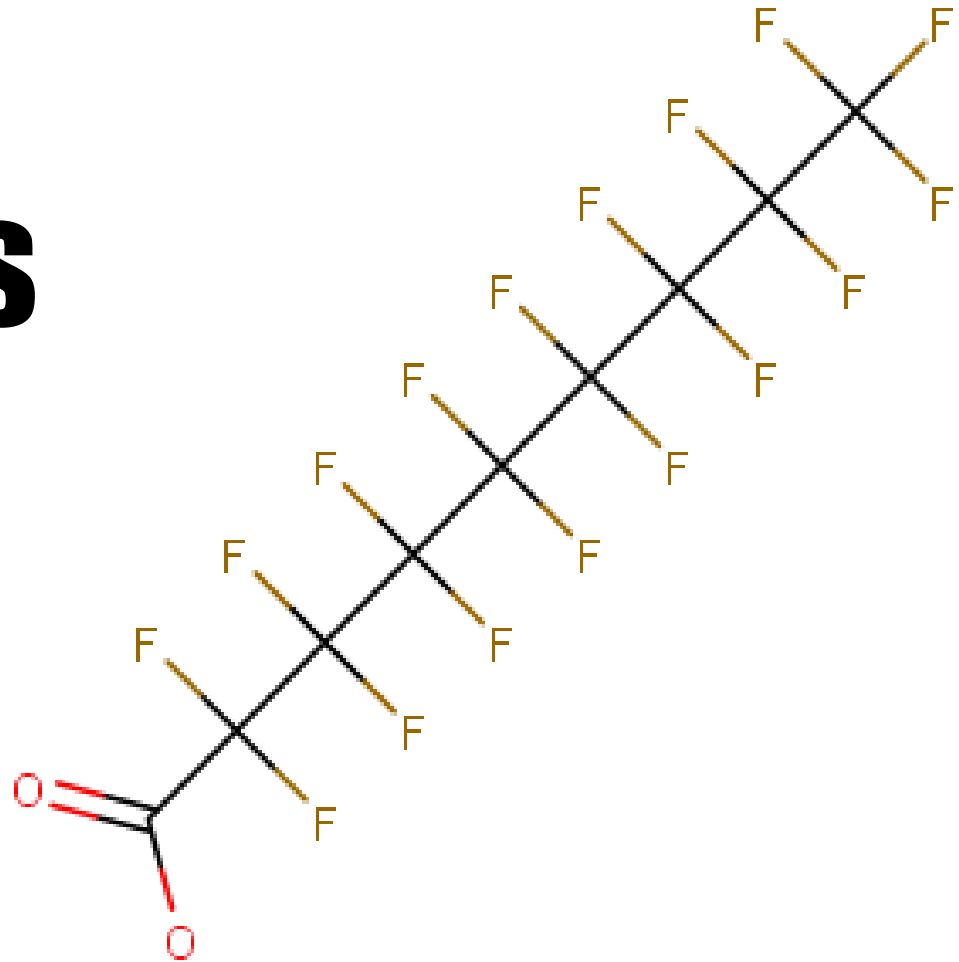
C13 INTERNAL STANDARD	%REC	RSD
PCB 1	3	54%
PCB 3	3	63%
PCB 4	7	36%
PCB 15	10	46%
PCB 19	15	45%
PCB 37	24	35%
PCB 54	20	26%
PCB 81	44	27%
PCB 77	47	28%

PCB 104	25	24%
PCB 123	44	25%
PCB 118	43	25%
PCB 114	44	25%
PCB 105	48	24%
PCB 126	55	25%
PCB 155	34	27%
PCB 167	47	26%
PCB 156	49	22%

PCB 157	51	25%
PCB 169	47	26%
PCB 188	39	23%
PCB 189	58	24%
PCB 202	45	25%
PCB 205	54	25%
PCB 208	51	25%
PCB 206	45	27%
PCB 209	45	26%



# LC-MS/MS Analysis of PFAS in soil



# Extreva Specs - PFAS

Test		Temp (°C)	Fill vol	Static phases	flow rate	control flow time	Extraction time	Comments
PFAS	<b>ASE</b>	100	50%	3 x 5 mins			1:36:00	as usual, nothing special
PFAS	<b>EXTREVA</b>	100	50%		5 mL/min	20mins	0:58:01	7076B on Channel 2 has low extracts ~30 mL instead of ~155 mL



# **Test Summary: PFAS → *soil***

Extreva

1. ASE350 method direct transfer



# LC-MS/MS of PFAS in soil

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## ASE 350

MPFBA	111	98	32
M5PFPeA	110	93	67
M5PFHxA	136	114	82
M4PFHpA	130	81	85
M8PFOA	104	100	95
M9PFNA	75	66	70
M6PFDA	97	90	89
M7PFUdA	83	92	90
MPFDoA	90	112	116
M2PFTeDA	61	95	91
M3PFBS	83	67	83
M3PFHxS	67	62	92
M8PFOS	118	98	101
M3HFPODA	87	78	64
D3NMEFOSAA	103	104	95
D5NETFOSAA	103	113	110
M8FOSA	85	64	75
M2-4:2FTS	52	37	50
M2-6:2FTS	61	55	87
M2-8:2FTS	103	79	104



# LC-MS/MS of PFAS in soil

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## EXTREVA

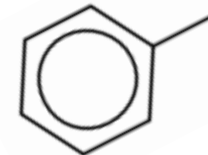
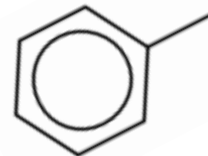
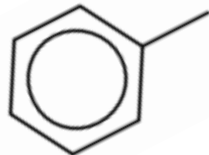
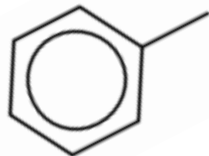
MPFBA	53	63	40
M5PFPeA	54	66	64
M5PFHxA	64	86	79
M4PFHpA	65	83	81
M8PFOA	77	102	93
M9PFNA	60	74	67
M6PFDA	74	95	88
M7PFUdA	64	86	79
MPFDoA	73	101	73
M2PFTeDA	65	93	74
M3PFBS	41	46	55
M3PFHxS	52	63	70
M8PFOS	77	101	92
M3HFPODA	43	8	94
D3NMEFOSAA	81	96	83
D5NETFOSAA	76	92	86
M8FOSA	59	87	69
M2-4:2FTS	24	21	30
M2-6:2FTS	46	51	61
M2-8:2FTS	64	73	76



# Extreva Specs

No. of samples	Extraction only time	
	Extreva	ASE350
4 samples	~50 minutes	~ 130 minutes
16 samples	~200 minutes	~ 520 minutes
Extraction Solvent	9:1 toluene: methanol	

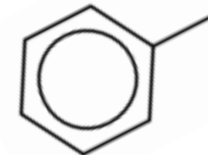
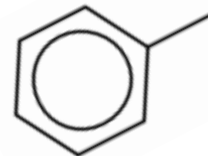
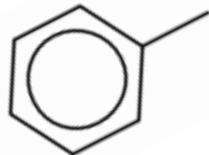
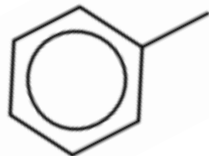
No. of samples	Extraction + Evaporation	
	Extreva	ASE350 + Rocket
4 samples	~171 minutes	~ 220 minutes
16 samples	~684 minutes	~ 620 minutes
Extraction Solvent	9:1 toluene: methanol	



# Extreva Specs

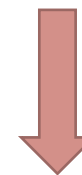
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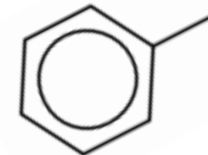
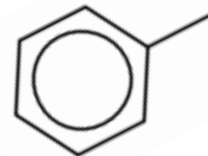
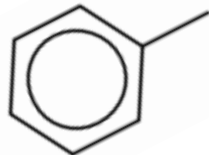
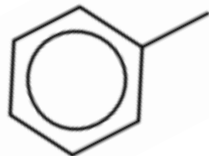
# Extreva Specs

Does not include manual labour for transferring samples from ASE350 to Rocket



No. of samples	Extraction only time	
	Extreva	ASE350
4 samples	~50 minutes	~ 130 minutes
16 samples	~200 minutes	~ 520 minutes
Extraction Solvent	9:1 toluene: methanol	

No. of samples	Extraction + Evaporation	
	Extreva	ASE350 + Rocket
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Extraction Solvent	9:1 toluene: methanol	





# Summary → METHOD DEVELOPMENT



# Summary → EXTRACTION TIME





# General Recommendations

- We are working to increase uptime; notification system
- Push the limit to reduce solvent usage; design of experiment
- Continue to test real-world samples (soil, solids, tissue) at random



# EXTREVA ASE Key Benefits



## Streamlined Sample Extraction

(Combines sample **extraction, clean-up and concentration**)



## Combined Footprint

(Extraction and Evaporation System in **one platform**)



## Increased Laboratory Productivity

(Parallel Extraction of up to 4 samples at one, **reduces cost** per sample)



## Complete Workflow from Sample to Vial

(True **walk-away** technology)

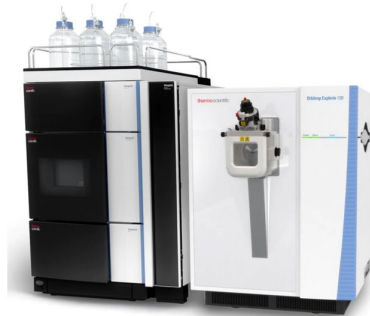
Increased productivity, better consistency & reduces costs

# EXTREVA™ ASE™ Integrated Analytical Workflows



Typical Applications:

- PAHs from soil
- PCBs from soil
- OCPs from soil



Applications:

- Extractables and Leachables in food contact materials

Automation from Sample to Vial Eliminates Pressures of Sample Preparation

# How does EXTREVA ASE helps you

## Sample prep pain points

- 1 Complicated workflows
- 2 Sample throughput
- 3 Labor intensive workflows
- 4 Risk of errors
- 5 High costs – Labor and solvent usage
- 6 Target analyte recovery and reproducibility
- 7 Sample tracking

## How EXTREVA ASE solves issues

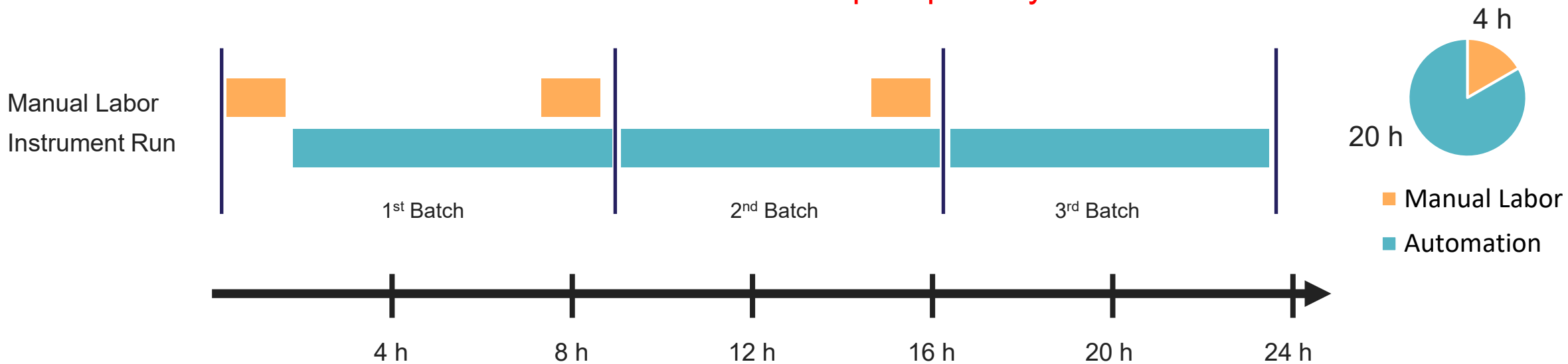
- 1 Accomplishes extraction and concentration without user interaction
- 2 Extract and concentrate 4 samples in parallel
- 3 Completes the entire process without the need for intervention
- 4 Methods are performed consistently without outside distractions
- 5 Reduces the required hands-on time and solvent used for extraction
- 6 Automation brings a higher level of consistency and accuracy over manual prep
- 7 2D barcode reading records sample prep parameters

Cost of Errors -%RSD, Reruns, Lost samples

# Extreva vs. ASE-350: 24-h Total Workflow (Extraction + Evaporation)

Based on 10-mL cell extraction

Extreva: 48 samples per day



ASE-350 + Rocket Evaporator: 36 samples per day

# EXTREVA ASE impact on your lab

*"I want to put my sample in one side and have it ready for analysis"*



**First of its kind!** Start to finish sample prep without user intervention



## Save time, increase productivity

- ✓ Parallel processing, combined automated extraction and evaporation without instrument interaction



## Control cost

- ✓ New gas assisted extraction saves on solvents and extraction time
- ✓ Automation of whole process reduces errors
- ✓ Training new analysts faster and easier



## Data integrity for accreditation

- ✓ 2D barcode tracking allows for full documentation of all sample conditions

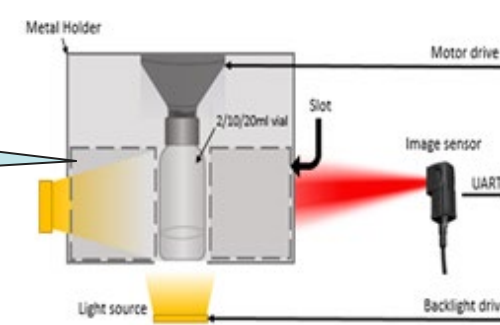
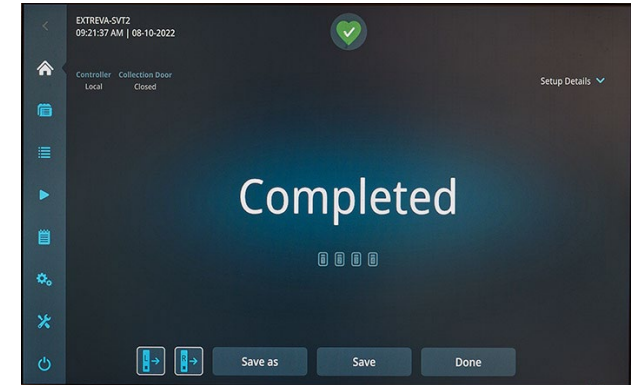
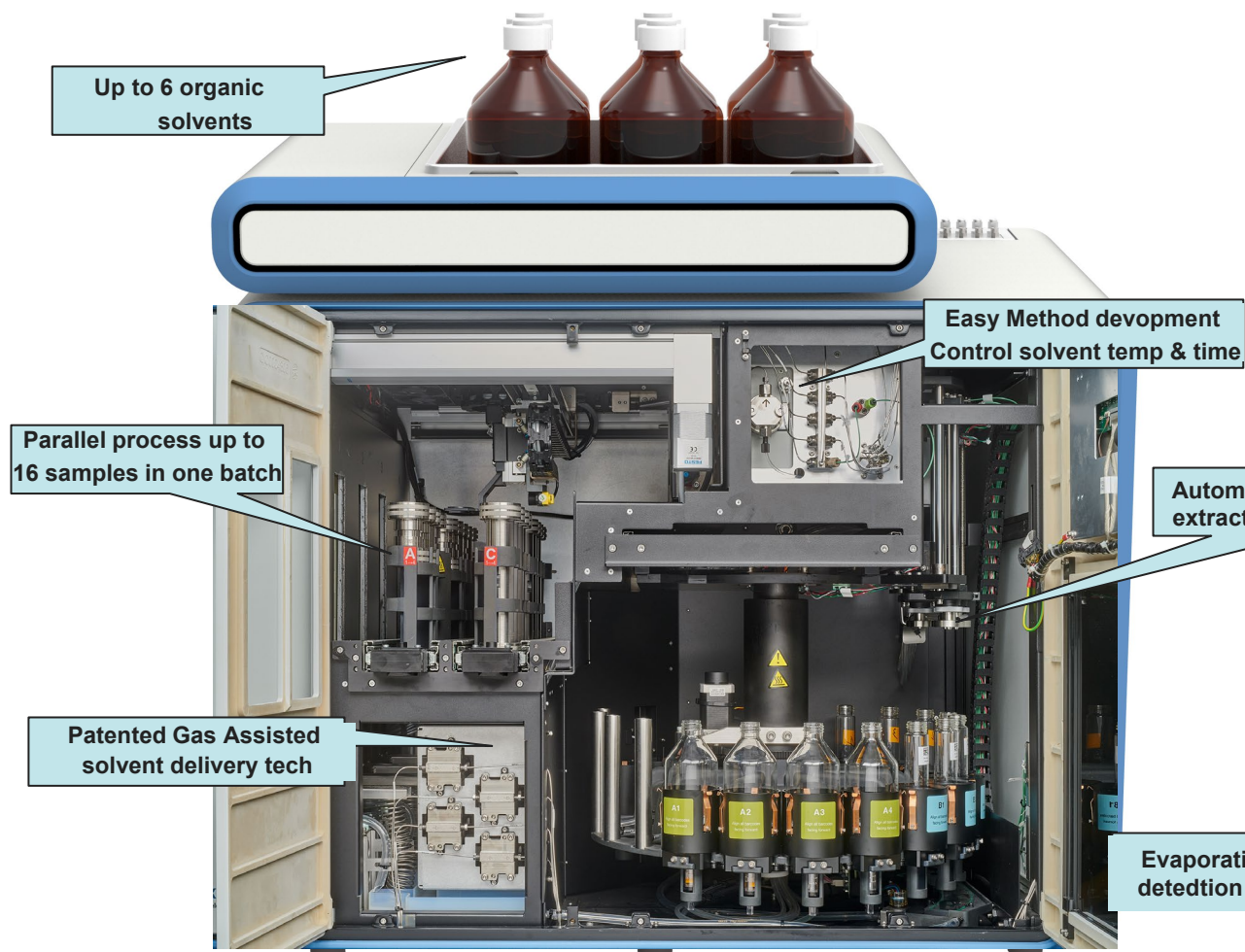


# Inside the **EXTREVA ASE system**

# Thermo Scientific- EXTREVA™ ASE™

Accelerated Solvent Extractor

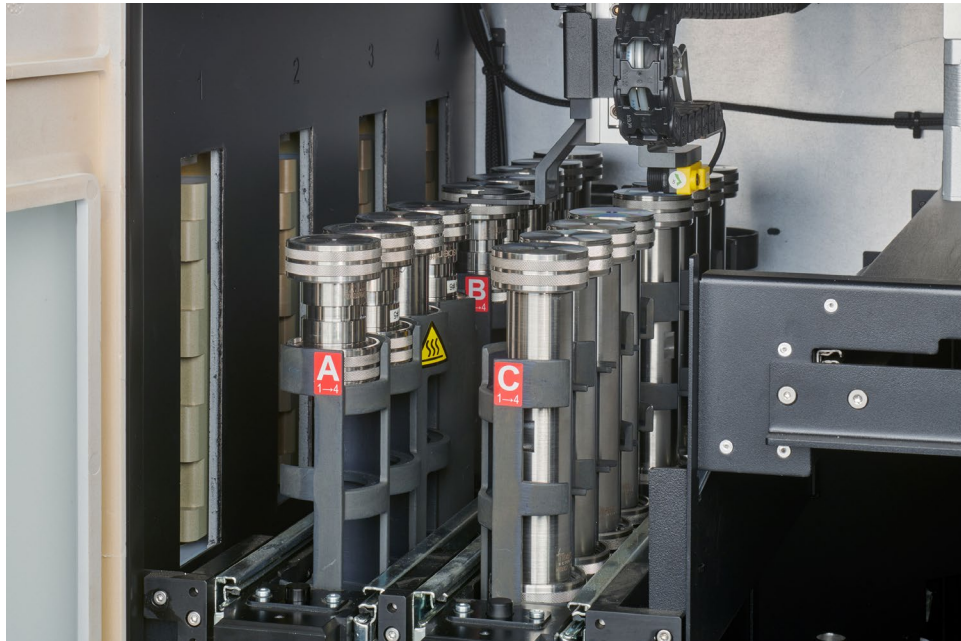
True Walkaway Automated Extraction & Concentration



# Parallel Processing

EXTREVA ASE delivers value:

- Increases lab throughput → Increases profitability
- Improves processing time → Reduced risk of missing hold times and helps lab bottom line



Features:

- Parallel process of 4 samples
- In cell clean up and moisture removal
- 16 sample autosampler – Same cell size per group of 4

# Extract collection and solvent evaporation

- Extracts are collected on 4 independent channels
- After collection or during the needles can be rinsed
- With the vessels still sealed, evaporation process starts automatically – no user intervention required
  - Gentle vacuum is applied to not lose semi volatile compounds
  - N<sub>2</sub> is blown through the same collection needle
  - Each bottle can be heated to help facilitate evaporation



# Combining extraction and evaporation

Why combine two operations?

- Allows for true walk-away sample prep
- Less user intervention required for the entire process
- Analyte can dedicate time to more important tasks
- Alleviates worry of errors, spills or the need for purchase and maintain multiple devices

Patented design:

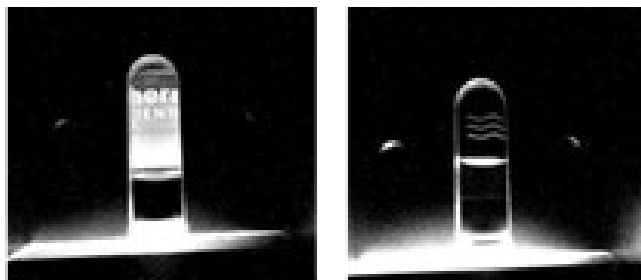
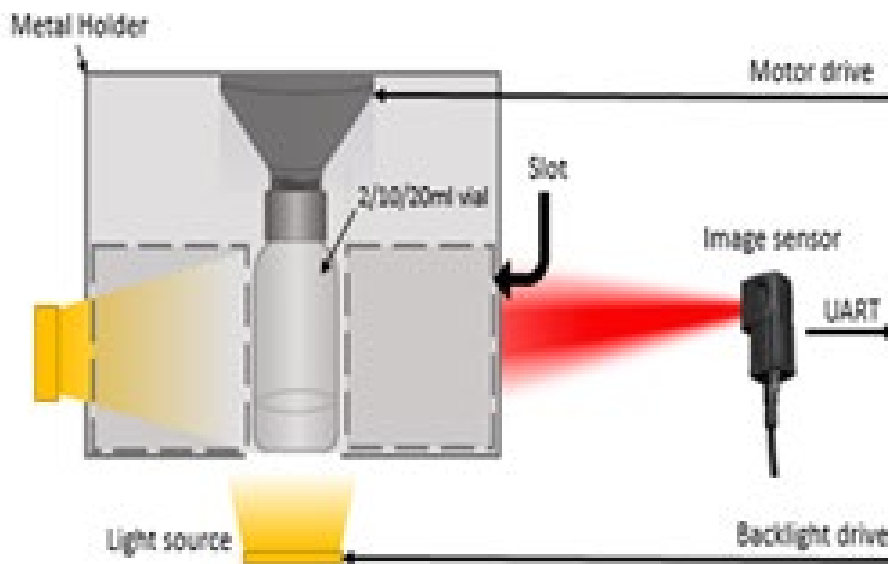
- Vacuum nitrogen connect directly to the collection vessels
- Combined mode helps facilitate evaporation
- Incorporate the GC or LC vial into the collection vessel



# Automated end-point detection

Monitoring of evaporation no longer required

- Allows for true walk away sample prep
- Each channel will stop at end point even if the others have not reached the end point



Automated end-point detection using machine learning solves this issue

- Using an image sensor and proper backlighting allows the instrument to get a real picture of the evaporation level
- Machine learning is employed to teach the instrument to stop at the process at the desired level

# Acknowledgements

**ThermoFisher**  
SCIENTIFIC

